

Sampling and testing carbon black for use in the rubber industry —

Part 12: Methods for determination of surface area by surfactant adsorption

ICS 83.040.20

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Committees responsible for this British Standard

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British Rubber Manufacturers' Association Ltd.

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National foreword

This Part of BS 5293 has been prepared by Technical Committee PRI/53 and is identical with ISO 6810:1995 *Rubber compounding ingredients — Carbon black — Determination of surface area — CTAB adsorption methods*, published by the International Organization for Standardization (ISO).

It supersedes BS 5293-12:1988 which is withdrawn.

Cross-references

Publication referred to	Corresponding British Standard
ISO 1126:1992	BS 5293 <i>Sampling and testing carbon black for use in the rubber industry</i> Part 5:1992 <i>Method for determination of loss of mass on heating</i> (Identical)
ISO 1304:1985	Part 10:1988 <i>Method for determination of iodine adsorption number</i> (Identical)
ISO 4652-1:1994	Part 11:1995 <i>Method for determination of specific surface area by nitrogen adsorption methods using single-point procedures</i> . (Identical)

The Technical Committee has reviewed the provisions of ISO 385-1:1984, ISO 648:1977 and ISO 6809:1989 to which normative reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard.

A related British Standard to ISO 385-1:1984 is BS 846:1985 *Specifications for burettes* and a related British Standard to ISO 648:1977 is BS 1583:1986 *Specification for one-mark burettes*.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies methods for the determination of the surface area of carbon blacks, excluding the area of micropores that are too small to admit molecules of hexadecyltrimethylammonium bromide (cetyltrimethylammonium bromide, commonly referred to as CTAB).

The methods are suitable for characterizing rubber grade carbon blacks of all types.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1126:1992, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*.

ISO 1304:1985, *Rubber compounding ingredients — Carbon black — Determination of iodine adsorption number — Titrimetric method*.

ISO 4652-1:1994, *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Part 1: Single-point procedures*.

ISO 6809:1989, *Rubber compounding ingredients — Carbon black — Standard reference blacks*.

3 Principle

3.1 The isotherm for adsorption on carbon black of CTAB from an aqueous solution has a long horizontal plateau corresponding to monomolecular coverage of the substrate surface from which the adsorbate is not sterically excluded. The CTAB adsorption by carbon black is not affected by tarry materials and functional groups containing hydrogen, oxygen, etc. Rapid equilibrium is achieved by using mechanical stirring and ultrasonic vibration. Titration with sodium di(2-ethylhexyl) sulfosuccinate solution to a maximum turbidity or colour end-point is used to determine the unadsorbed CTAB after removal of the colloiddally dispersed carbon black by filtration. All results are determined relative to a reference black, the CTAB surface area of which is assumed to be exactly as specified in ISO 6809.

3.2 Titration of the unadsorbed CTAB is carried out using one of the following methods:

- a) method 1, using sodium di(2-ethylhexyl) sulfosuccinate solution by automatic titrimeter to a maximum-turbidity end-point;
- b) method 2, using sodium di(2-ethylhexyl) sulfosuccinate solution by manual titration to a maximum-turbidity end-point;
- c) method 3, using sodium di(2-ethylhexyl) sulfosuccinate solution by manual titration to a specified colour end-point;
- d) method 4, using sodium dodecyl sulfate (SDS) solution by manual titration to a specified colour end-point.

4 Reagents

All reagents shall be of recognized analytical grade. Distilled water, or water of equivalent purity prepared by passing it through a fixed bed of ion-exchange materials, shall be used. The purified water shall be stored in suitable vessels, and transfer tubing shall be made of polytetrafluoroethylene, polyethylene, quartz, or other materials resistant to chemical attack.

4.1 Buffer solution, of pH 7.

Dissolve 2,722 g of potassium dihydrogen orthophosphate (KH_2PO_4), 4,260 g of disodium hydrogen orthophosphate (Na_2HPO_4) and 1,169 g of sodium chloride (NaCl) in water and dilute to 1 dm³.

NOTE 1 Further details concerning the preparation are given in BATES et al., *J. Res. NBS*, **29** (1942), p.183. An equivalent buffer solution is available commercially.